OPTIMISATION OF TORREFACTION AND PELLETISATION CONDITIONS FOR DURIAN SEED BIOMASS

Nur Alya Maisarah Mohd Khalid, Nor Adilla Rashidi*

Department of Chemical Engineering, HICoE Centre for Biofuel and Biochemical Research, Universiti Teknologi PETRONAS, Perak, Malaysia

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ABSTRACT

Torrefaction of biomass is an effective thermochemical pre-treatment technology that improves biomass characteristics for solid fuel (bio-coal) generation. The abundance of durian seeds in Malaysia led to their selection due to their high energy potential. A comparison was made between the effects of torrefaction temperatures (200°C, 250°C, and 300°C) and residence periods (30, 45, and 60 min at 250°C) on mass, energy yield, and calorific value. Torrefaction was carried out in a Protherm tube furnace under inert conditions, with a heating rate of 10°C/min and a nitrogen flow of 0.2 L/min. The results reveal that 250°C for 30 min produces the best calorific value (22.529 MJ/kg) while also maintaining high mass (23.2 wt.%) and energy yield (31.34%). Proximate analysis, surface chemistry, and thermogravimetric analysis were utilised to investigate the properties of raw and optimised biomass. Torrefaction decreased moisture and volatile matter while raising fixed carbon and ash levels. The optimum sample had 3.24 wt.% moisture, 62.93 wt.% volatile matter, 17.79 wt.% fixed carbon, and 16.04 wt.%. The impact of pelletisation was also investigated, with various moisture contents (5 wt.%, 10 wt.%, and 15 wt.%) impacting the process. At 5 wt.% moisture, the maximum durability (98.79%) and calorific value (22.956 MJ/kg) were obtained. Overall, the study validates the effectiveness of durian seed biomass as a feasible renewable energy source.

Keywords: *Durian see, palletisation, pellet biomass, durability, calorific value, mass yield, energy yield*

INTRODUCTION

The growing global demand for energy has heightened the need for sustainable energy solutions, particularly in Malaysia, where reliance on coal for electricity generation is exacerbating environmental issues, such as rising carbon dioxide (CO_2) emissions [1]. Despite continued efforts to reduce emissions, Malaysia remains heavily reliant on fossil fuels, with coal accounting for the majority of the country's CO_2 emissions. Hence, biomass has emerged as a promising alternative to reduce CO_2 emissions, due to its renewable nature [2]. However, constraints such as low energy density, high moisture content, and poor transportability hinder the large-scale use of the pristine biomass in energy generation. Specifically, these limitations result in high transportation costs and lower energy output, rendering pristine biomass inefficient for practical energy use. Hence, torrefaction, a thermochemical process in which biomass is heated at moderate temperatures (200-300°C) in an oxygen-deficient environment, has been extensively studied as a method to improve biomass properties, such as energy density, grindability, and hydrophobicity [3]. As such, these enhancements will enable the torrefied biomass to be applied in combustion as well as other bioenergy applications. Additionally, the pelletisation process will transform the torrefied biomass into a homogeneous and compact pellet, thereby simultaneously improving its energy density, transportability, and storage stability [4].

Primarily, the torrefaction efficiency is strongly reliant on temperature, residence duration, and moisture content, all of which influence the degradation of biomass components such as hemicellulose, cellulose, and lignin [5]. Therefore, this study focuses on optimising the torrefaction parameters of durian seed, specifically temperature and residence time, to increase the energy yield and durability of durian seed biomass. Specifically, the use of durian seeds, which constitute 20-25% of the fresh fruit, is often discarded through landfilling or left on-site to decompose. To the best of our knowledge, while the torrefaction process has been extensively studied for durian peel and shell, the utilisation of durian seeds remains largely underexplored. In line with the findings of Chen et al. [6], variations in feedstock selection can lead to different outcomes, hence emphasising the importance of investigating the potential of durian seed biomass in this study. Furthermore, the influence of pelletisation, which enhances transportability and durability, will be examined to solve logistical issues in bioenergy applications [4]. Overall, it is anticipated that integrating the torrefaction and pelletisation processes of durian seed biomass could improve its mechanical strength, thermal stability, and degradability, thereby increasing its viability as a sustainable energy source and reducing Malaysia's reliance on coal.

*Corresponding author.
E-mail address : adilla.rashidi@utp.edu.my

METHODOLOGY

Pre-treatment

Durian seeds were collected from Seri Iskandar, Perak, weighing a total of 6 kg. The seeds were pre-treated by washing to remove impurities, then cut into pieces (1-3 cm). They were air-dried for 24 hours and then oven-dried at 105°C for 24 hours, until a moisture content of below 10 wt.% was achieved. The dried seeds were ground using a biomass grinder and sieved to a uniform size of 0.5 mm using a sieve shaker. The prepared biomass was stored in a sealed container to prevent moisture absorption.

Durian Seed Torrefaction Process

The durian seed biomass was torrefied using the Protherm tube furnace under an inert nitrogen atmosphere, with a fixed flow rate of 0.2 L/min and a heating rate of 10°C/min [7]. In terms of the effect of temperature, 20 g of prepared biomass was placed in the furnace and torrefied at 200°C, 250°C, and 300°C, with a constant residence period of 30 min. Subsequently, to examine the influence of the residence period, the torrefaction process was conducted for 30, 45, and 60 min at a constant temperature of 250°C. Upon each torrefaction session, the samples were cooled to ambient temperature and weighed to calculate the mass yield, which indicates the degree of thermal degradation.

Effect of Pelletisation

The moisture content of the sample was adjusted to 5 wt.%, 10 wt.%, and 15 wt.% to evaluate its effect on pellet durability. According to Pradhan et al. [8], moisture in biomass acts as a binder during pelletisation, enhancing bonding through the van der Waals forces. The optimal moisture content for pelletisation typically ranges from 10% to 15% [5], depending on the type of biomass. The sample was then compressed under 1 ton of pressure for 10 min using a 13 mm steel mould and hydraulic press. The amount of water added was calculated using [9]:

$$W_w = W_s \times \frac{(m_f - m_i)}{100 - m_f} \quad (1)$$

Referring to Equation 1, W_w refers to the amount of water to be added (g), W_s is the initial moisture content (g), m_i is the initial moisture content (% by wet basis), and m_f is the final moisture content (% by wet basis).

Characterisations

Mass and Energy Yield

Mass yield indicates the degree of thermal decomposition, while energy yield reflects the efficiency of the torrefaction process by accounting for both mass reduction and energy content increase [10]. The mass yield and energy yield of the samples were calculated using [9]:

$$\text{Mass yield (\%)} = \frac{\text{Mass after torrefaction}}{\text{Mass of raw sample}} \times 100\% \quad (2)$$

$$\text{Energy yield (\%)} = \frac{\text{Mass yield} \times \text{HHV}_{\text{torrefiedDS}}}{\text{HHV}_{\text{raw DS}}} \times 100\% \quad (3)$$

Calorific Value and Higher Heating Value (HHV)

HHV was determined using an oxygen bomb calorimeter to assess the energy density of the biomass [11].

Proximate Analysis

The proximate analysis includes moisture content (MC), volatile matter (VM), ash content (AC) and fixed carbon content. The mathematical equations utilised are expressed as:

$$\text{MC}(\%) = \frac{W_i - W_f}{W_i} \quad (4)$$

$$\text{VM}(\%) = \frac{W_i - W_f}{W_i - W_c} \quad (5)$$

$$\text{AC}(\%) = \frac{W_3 - W_1}{W_2 - W_1} \quad (6)$$

$$\text{Fixed carbon (\%)} = 100\% - (\text{MC}(\%) + \text{VM}(\%) + \text{AC}(\%)) \quad (7)$$

The moisture content was determined by heating a 2 g sample to 105°C in an OHAUS moisture analyser until a consistent mass was achieved. The volatile matter content was determined by heating 0.5 g of biomass in a crucible to 950°C for 7 min in a muffle furnace [12], as per ASTM E872-82. The weight loss was then used to calculate the percentage of volatile matter. Ash content was evaluated by heating the biomass sample in a muffle furnace at 500-600°C for 3 hours and estimating the residue left after oxidation, as per ASTM E872-82 [12]. After determining the MC, VM, and AC as a percentage of the total (100%), the residue was estimated as the fixed carbon content [11]-[12].

Referring to Equation 4, W_i and W_f represent the mass of the sample (g) before and after drying to constant weight [13]-[14]. While for Equation 5, W_i, W_f, and W_c refer to the initial weight of the crucible with lid containing samples before heating (g), final weight of the crucible with lid containing samples after heating (g), and weight of the crucible with lid (g), respectively [15]-[16]. Lastly, for Equation 6, W_1, W_2, and W_3 refer to the weight of the empty crucible (g), the weight of the crucible and sample before heating (g), and the weight of the crucible with ash residue after heating (g) [13],[15].

Thermogravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR)

TGA was performed using a thermogravimetric analyser to assess the thermal stability and decomposition behaviour of the biomass [10], which enabled the determination of the optimal temperature range for torrefaction. In TGA, both pristine and torrefied samples (at optimum conditions) were heated from room temperature up to 800°C under an inert nitrogen flow. Furthermore, the chemical composition of raw and torrefied biomass was analysed using FTIR at a spectral range of 4000-400 cm^{-1}, which identified functional groups associated with cellulose, hemicellulose, and lignin, such as hydroxyl (-OH), carbonyl (C=O), and aliphatic (C-H) groups.

Durability Analysis

Pellet durability was evaluated using a drop test, where pellets were dropped from a height of 1 m on a concrete surface, and the mass of the unbroken pellet was used to calculate durability [17]. These tests were performed on samples with varying moisture contents (5%, 10%, and 15%) to determine the optimal conditions for biomass conversion and storage. Furthermore, the durability of the durian seed pellet can be calculated using [17]:

$$\text{Durability}\,(\%) = \frac{\text{Weight of pellet after drop}}{\text{Weight of pellet before drop}} \times 100\% \qquad (8)$$

RESULTS AND DISCUSSIONS

Analysis of Non-Torrefied and Torrefied Samples

Mass and Energy Yield

In the torrefaction process, both operating temperature and retention time are the predominant factors that control the intensity of the thermal treatment [18]. From Table 1, it is observed that as temperature and residence time increased, mass yield and energy yield decreased, indicating a progressive thermal degradation of biomass components. Specifically, in the context of temperature at a constant residence time of 30 minutes, the mass yield of torrefied durian seed exhibits a declining trend, ranging from 31.81 wt.% at 200°C to 11.55 wt.% at 300°C. Referring to Akhtar et al. [19], sample decomposition during the torrefaction process is associated with the release of oxygen-containing molecules, including water, CO_2, and carbon monoxide. Additionally, Mamvura and Danha [20] reported that at a temperature of approximately 210°C, light torrefaction occurs, where only hemicellulose is degraded, thereby retaining the maximum amount of mass and energy of the biomass. Then, at a moderate temperature of around 250°C, mild torrefaction occurs, during which cellulose degradation also takes place. Lastly, at a higher temperature of 290°C, severe torrefaction is expected, where the decomposition of lignin, cellulose, and remaining hemicellulose is anticipated, leading to the highest mass and energy loss. Likewise, under the similar residence time, the energy yield decreased in a similar trend, from 39.33 wt.% at 200°C to 12.17 wt.% at 300°C. Such findings are anticipated since the energy yield calculation is dependent on the mass yield value. Herein, the reduction in energy retained after the torrefaction process at high temperature is due to the release of volatile matter, which initially contains some of the fuel's energy.

Moreover, both mass yield and energy yield gradually decrease as the residence times increase from 30 min to 60 min, a trend comparable to that observed when the durian seed samples are subjected to rising temperatures. Theoretically, longer residence times allow for more severe dehydration and carbonisation to occur. These findings are further supported by Orisaleye et al. [21], who observed that the highest mass loss occurred at the maximum operating conditions of 280°C and 90 min. Nevertheless, the energy yield that decreases less sharply compared to the mass yield implies that the energy has been effectively concentrated in the torrefied product. Overall, identifying the optimal settings for durian seed biomass is crucial to striking a balance between preserving energy output and enhancing biomass quality, while minimising excessive charring that results from severe thermal degradation.

Calorific Value and Higher Heating Value (HHV) Analysis

The raw durian seed exhibited an HHV of 16.680 MJ/kg, which was relatively low compared to other raw biomass. Briefly, Jiang et al. [22] reported that greater moisture content will affect the HHV, as more energy is consumed to evaporate the water during combustion. Referring to Table 2, the HHV increased with temperature at a fixed residence time, reaching 20.626 MJ/kg at 200°C and 22.529 MJ/kg at 250°C, but dropping to 17.587 MJ/kg at 300°C. This drop is caused by the degradation of lignin at higher temperatures, which disturbs its aromatic structure and reduces its ability to increase energy density [23]. As the residence time was varied at a constant 250°C, the HHV initially increased at 30 min (22.529 MJ/kg), but declined with longer residence times,

Table 1 Mass yield and energy yield with respect to variation in temperature and residence time

Temperature (°C)	Residence Time (min)	Initial Mass (g) (g)	Final Mass (g)	Mass Yield (wt.%)	Energy Yield (%)
200	30	20	6.361	31.81	39.33
250	30	20	4.640	23.20	31.34
	45	20	2.479	12.40	13.45
	60	20	1.992	9.96	10.19
300	30	20	2.309	11.55	12.17

Table 2 HHV and its increment for torrefied durian seed

Parameter		HHV (MJ/kg)	Increment in HHV (%)	Energy Densification Ratio (IDR)a
Raw Durian Seed		16.680	-	-
Torrefaction	200°C/ 30 min	20.626	23.66	1.24
	250 °C/ 30 min	22.529	35.07	1.35
	250 °C/ 45 min	18.093	8.47	1.08
	250 °C/ 60 min	17.063	2.30	1.02
	300 °C/ 30 min	17.587	5.44	1.05

Note: Energy densification ratio (IDR) = $HHV_{product}$ / $HHV_{feedstock}$

reaching 18.093 MJ/kg at 45 min and 17.063 MJ/kg at 60 min. This reduction is consistent with Dirgantara et al. [24], who proposed that the optimal residence time for torrefaction is 20-40 min, as longer heating times cause the breakdown of lignin and cellulose, resulting in volatile losses. Considering that the effectiveness of the torrefaction process is determined by the mass yield, energy yield, and energy diversification ratio [18], it can be concluded that torrefaction at 250°C for 30 min was the most effective for durian seeds. Besides, this heating value is considered acceptable when compared to other torrefied biomass, with discrepancies related to variations in the biomass's hemicellulose, cellulose, and lignin compositions.

Proximate Analysis

Table 3 shows that the torrefied durian seed had less moisture and volatile matter than the raw sample, as the torrefaction process removed water and decomposed hydrogen- and oxygen-containing organic compounds, releasing volatile chemicals and producing a hydrophobic biomass [6]. The moisture level of raw durian seed (6.38%) was lower than that reported by Haryati et al. [25] (8.71%), most likely due to variance in durian seed varieties. The ash percentage of the raw sample increased significantly from 4.25% to 16.04% during the torrefaction process, in agreement with Pimsamarn et al. [10]. This increase in ash content is related

Table 3 Proximate analysis (average) of raw and torrefied durian seed at 250°C/30 min

Parameter	Moisture (%)	Volatile matter (%)	Ash (%)	Fixed carbon (%)
Raw Durian Seed	6.38	78.80	4.25	10.57
Torrefied durian seed	3.24	62.93	16.04	17.79

to the elimination of organic volatiles during devolatilisation, which leaves behind inorganic residues that might reduce the combustion efficiency [26]. Nevertheless, further carbonisation enhances both the carbon content and stability. Specifically, the fixed carbon content notably increased from 10.57% to 17.79%, as carbonisation increases carbon by eliminating oxygen and hydrogen atoms, ultimately leading to a high calorific value.

Thermogravimetric Analysis

According to Barzegar et al. [27], the thermal breakdown of lignocellulosic biomass happens in three stages (Figure 1). Previous research has shown that hemicellulose degrades between 210°C and 320°C, cellulose between 320°C and 399°C, and lignin between 150°C and 899°C [28]. Stage I, which occurs up to 200°C, is the evaporation of absorbed and bonded water in the biomass. The raw durian seed lost 5.77% of its weight, whereas the torrefied

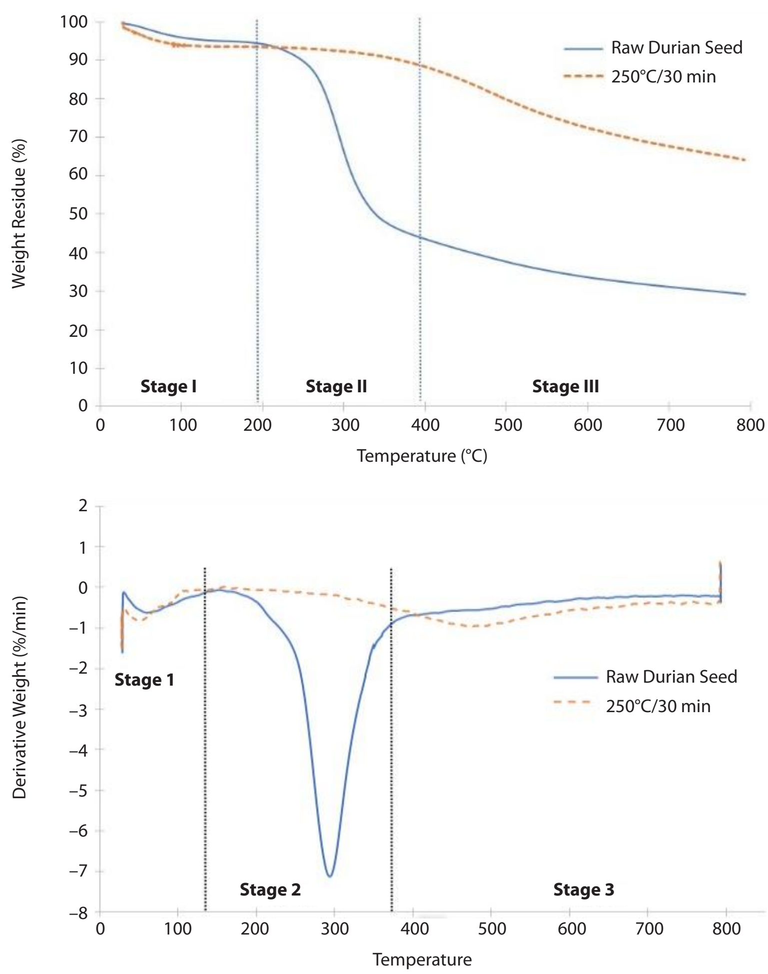

Figure 1 TGA and DTG curves of raw and torrefied durian seed at 250°C and 30 min

sample lost 6.54% after being heated to 250°C for 30 min. Despite torrefaction reducing moisture content, the torrefied sample most likely absorbed moisture from the environment during the two weeks preceding the examination, which explains the observed results. In Stage II (200-400°C), the raw sample lost 50.73% of its weight, whereas the torrefied biomass lost only 5.14% due to the partial degradation of hemicellulose and cellulose during torrefaction. The significant weight loss in the raw sample is due to the heat degradation and devolatilisation of volatile materials, particularly hemicellulose and cellulose, which leave behind fixed carbon. This is demonstrated by the DTG curves, which indicate a more pronounced peak at Stage II for the raw sample, comparable to the studies by Ivanovski et al. [29] on the torrefaction of miscanthus and hop waste. Stage III, which occurs at temperatures exceeding 400°C, is characterised by the breakdown of lignin and the combustion of inorganic elements, leading to ash production. Lignin depolymerises, resulting in aromatic chemicals and gases, including CO_2 and methane [28]. Finally, the raw biomass left a lower residual mass than the torrefied sample, indicating that the torrefied biomass had a higher ash content due to the concentration of inorganic minerals after organic matter was removed during torrefaction.

FTIR Analysis

FTIR analysis was performed on both raw and optimally torrefied durian seed (250°C for 30 min) to investigate the functional groups contained in the biomass, using a wavenumber range of 4000 to 400 cm^{-1}. The prominent peak between 3200 and 3600 cm^{-1} represents O-H stretching [17], primarily from lignocellulosic components such as lignin and cellulose, which is attributed to the presence of water and hydroxyl groups. This peak decreased in the torrefied sample due to a decrease in moisture content and carbohydrate degradation during torrefaction [30]. Peaks between 2850-2950 cm^{-1} [17], which correspond to C-H stretching bonds in lignin and cellulose, appear less sharp in the torrefied sample, implying the partial breakdown of the sample upon torrefaction. Ong et al. [31] reported that the peak at 1459 to 1596 cm^{-1} is related to C = C stretch, mainly due to stretching and skeletal vibrations of aromatic rings. Herein, its appearance in the torrefied sample justifies the increment in carbon content as well as the calorific value. Furthermore, the peaks within the range of 1850-1600 cm^{-1} refer to the carbonyl/carboxyl and ester groups in cellulose and hemicellulose. Comparing the raw and torrefied durian seed, the disappearance of this peak indicates a complete decomposition of hemicellulose, a biopolymer known for its reactivity due to its low crystallinity and polymerisation degree [30]. Likewise, smoother and attenuated peaks in the range of 1200-900 cm^{-1}, corresponding to O-H bending, imply the decomposition of cellulose and lignin in the sample during the heating process. Overall, the summary of surface chemistry is summarised in Table 4.

Durability and Calorific Value Test on Pelletised Sample

Durability and calorific value tests are performed on pelletised raw, controlled, and optimal torrefied durian seed samples at 250°C for 30 min with varying moisture contents of 5%, 10%, and 15% to determine the optimal moisture content for both durability and calorific value, ensuring suitability for energy applications and storage (Table 5). Durability is crucial in deciding pellet quality, as it evaluates the pellet's ability to withstand physical disintegration during transit and handling. The highest durability was obtained with a 10% moisture content (99.07%), followed by 5% moisture (98.79%) and the controlled samples (98.63%), while the 15% moisture content had the lowest durability (69.42%). The 15% moisture sample had a substantially greater standard deviation, indicating inconsistent results, which were most likely caused by the sample's brittleness. The ideal moisture content strikes a compromise between durability and calorific value, with increased moisture enhancing durability by up to 10% before declining at 15%. This is because raw durian seeds, which have not been thermally treated, have a higher cellulose and hemicellulose content, making them more prone to disintegration under mechanical pressure [32]. Excessive moisture weakens the pellet's bonds and reduces its integrity, as additional water accumulated on the particles' surfaces prevents pellet formation [22],[33].

HHV which is required for pellet application, was highest for the torrefied sample at 5% moisture (22.956 MJ/kg), followed closely by the controlled sample (22.953 MJ/kg), 10% moisture content (21.688 MJ/kg), 15% moisture content (21.405 MJ/kg), and finally raw durian seed (16.326 MJ/kg). At optimal conditions, the non-pelletised samples had a lower calorific value than the pelletised samples, most likely because the pelletised samples have a higher bulk density, which improves combustion efficiency. Furthermore, decreased moisture concentration improves calorific value by increasing combustion efficiency [32], with 5% moisture content being ideal for both high calorific value and durability. In summary, the optimal moisture content for torrefied durian seed at 250°C for 30 minutes (5%) aligns with European standards for pellet moisture content (<10%) and durability (>95%) [17].

Table 4 FTIR main spectrum of raw and torrefied durian seed at 250°C and 30 min [17],[31]

Bond	Possible polymer	Raw durian seed	Torrefied durian seed	Wavelength
O - H	Alcohol, phenol (cellulose and lignin)	1023	1115	900 – 1200
C - H	Alkanes (cellulose)	1434	1374	1371 – 1382 1426 – 1465
C = C	Aromatic skeletal vibrations of lignin	-	1583	1459 – 1596
C = O	Carbonyl/carboxyl and ester groups (cellulose, hemicellulose)	1631	-	1600 – 1850
C - H	Aliphatic/alkyl (lignin)	2927	2917	2850 – 2950
O - H	Alcohol, phenol, lignin	3430	3402	3200 – 3600

Table 5 Durability and calorific value of raw and torrefied durian seeds' pellet

Sample	Durability (%)	HHV (MJ/kg)
Raw durian seed	93.928	16.326
Control sample	98.626	22.953
Pellet with 5% sample moisture	98.792	22.956
Pellet with 10% sample moisture	99.070	21.688
Pellet with 15% sample moisture	69.422	21.405

CONCLUSIONS

The study on the torrefaction of durian seed biomass revealed that temperature and residence time have a substantial impact on mass yield, energy yield, and heating value, resulting in pellets that meet the international requirements. At 250°C and a 30-minute residence period, the maximum calorific value (22.529 MJ/kg) was achieved, representing a 35.07% increase over raw biomass and indicating an enhanced energy potential. However, as temperature and residence time increased, mass and energy yields decreased even further. Torrefaction enhances the thermal stability of biomass, as demonstrated by TGA studies. Moreover, upon compositional analysis, the findings indicate that the torrefied durian husk has a lower volatile matter and moisture content, while exhibiting higher fixed carbon and ash content. Pelletisation, particularly at 5% moisture, increased its durability (98.79%) and calorific value (22.95 MJ/kg), making it ideal for energy applications. Nevertheless, further investigation into other characteristics, such as particle size, composition, and flow rate of carrier gas, may also enhance the energy yield and ash content of the torrefied product. Similarly, the characteristics of bio-pellets are not solely dependent on moisture content. Still, they should also be examined with respect to variations in die temperature, pressure, holding time, and the use of binding agents. In addition, investigating the combination of durian seed and durian husk could provide further benefits for Malaysia, particularly given their vast abundance and existing challenges with disposal. Lastly, the CHNS analysis is recommended to accurately quantify the carbon, hydrogen, and nitrogen content, providing a more precise assessment of heating value and stability.

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